Voluntary - Public

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China - Peoples Republic of

Post: Beijing

National Dairy Standard - B6

Report Categories:

FAIRS Subject Report

Approved By:
Mark Petry
Prepared By:
Wu Bugang

Report Highlights:
On November 20, 2009, China notified the WTO of "National Food Safety Standard of the People's Republic of China for Determination of Vitamin B6 in Infant Foods and Dairy Products" as SPS/N/CHN/157. This standard relates to the quality specifications of milk. The date for submission of final comments to the WTO is January 1, 2010. The proposed date of entry into force has not been specified.

Executive Summary:
On November 20, 2009, China notified the WTO of "National Food Safety Standard of the People's Republic of China for Determination of Vitamin B6 in Infant Foods and Dairy Products" as SPS/N/CHN/157. This standard relates to the quality specifications of milk. The date for submission of final comments to the WTO is January 1, 2010. The proposed date of entry into force has not been specified.

Thanks go to the consortium of industry and 3rd country Embassies in Beijing for their assistance in translating and reviewing this standard.

This report contains an UNOFFICIAL translation of National Standard on the Determination of Vitamin B6 in Infant Foods and Dairy Products.

General Information:

BEGIN TRANSLATION

GB National Food Safety Standard
GB ××××—××××

Determination of Vitamin B6 in Infant Foods and Dairy Products

Draft for Comment

Issued on xx-xx-xxxx Implemented on xx-xx-xxxx

Issued by the Ministry of Health
of the People's Republic of China

1 Scope

This standard provides for Determination of Vitamin B6 in Infant Foods and Dairy Products.

This standard applies to the Determination of Vitamin B6 in Infant Foods and Dairy Products.

The detection limit of this standard: pyridoxine 15ug/kg, pyridoxal 13ug/kg, pyridoxamine 16ug/kg

2 Normative references

Through this standard, the terms of the following documents have become the terms of this standard. Note the date of all reference files, all modify versions (exclude the contents of the corrigendum) or revisions shall not apply to this standard file. For undated reference documents, the latest version applies to this standard.

Test method and using water is regulated in GB/T 6682.

3 Principle

After extracted with hot water, etc. sample is separated by C-18 chromatogram column and determined with a fluorescence detector and measured Vitamin B6 (pyridoxine, pyridoxal, and pyridoxamine) content by external standard method.

4 Reagents and materials:

If no special illumination, all reagents mentioned in this method are AR grade and water is regulated in GB/T 6682.

4.1 Amylase: enzymatic activity ≥1.5 U/mg
4.2 Octane sulfonate: GR
4.3 Galacial acetic acid: GR
4.4 Anhydrous methanol: chromatography
4.5 Triethylamine: chromatography
4.6 HCL solution: Conc. 5.0mol/L,0.1mol/L
4.7 NaOH solution: Conc. 5.0mol/L,0.1mol/L
4.8 Standard Solution
4.8.1 Vitamin B6 (pyridoxine, pyridoxal, pyridoxamine) stock standard solution, Conc. 1mg/ml

Accurately weigh about 0.05g Vitamin B6 standard substance to 50mL volumetric flask, dissolved with water, and add water to the mark.

4.8.2 Vitamin B6 (pyridoxine, pyridoxal, pyridoxamine) middle standard solution, concentration 20μg/mL

Pipette 1ml stock standard solution(4.8.1) to 50ml volumetric flask, and add water to the mark.

4.8.3 Vitamin B6 (pyridoxine, pyridoxal, pyridoxamine) working standard solution, concentration 0.4μg/mL

Pipette 1ml middle standard solution (4.8.2) to 50ml volumetric flask, and add water to the mark.

5 Instrument and Equipment

5.1 Ultrasound oscillator
5.2 HPLC chromatography: equipped with fluorescence detector
5.3 Analytical balance: accurate to 0.1mg
5.4 PH meter

6 Analysis procedure

6.1 Sample Pretreatment

6.1.1Amylun Sample

For solid sample, weigh about 5.0g; for liquid sample should mix equality, weigh about 20g(accurate to 0.1mg), to a 150mL conical flask, add about 0.5g α-amylase, then add 25mL of 45-50Celsiuswater and mix thoroughly. Fill nitrogen to the conical flask and seal it, placed it into 45Celsiusoven for 30min, then cool it to room temperature.

6.1.2 No asylum Sample

For solid sample, weigh about 5.0g; for liquid sample should mix equality, weigh about 20g (accurate to 0.1mg), to a 150mL conical flask, add 25mL 60Celsiuswater and mix to dissolve thoroughly, stand 5~10min, then cool it to room temperature.

6.2 Sample solution preparation

6.2.1 Slowly adjust PH of it to 1.70 with hydrochloric acid solution (4.6), let it stand for 1min, then adjust pH of it to 4.50 with sodium hydroxide (4.7)

6.2.2 Transfer the sample solution into a 50mL volumetric flask; wash the conical flask with distilled water repeatedly and combine the washing liquid to the 50mL volumetric flask then add water to the mark.

6.2.3 place above-mentioned conical flask to ultrasonator (5.1) for shaking 10min

6.2.4 To take the triangle funnel with filter paper, above another 50ml volumetric flask, pour sample solution into filter paper, filter the solution through filter paper. Again filter the solution through 0.45μm membrane filter and collect the filtrate as injection sample.

6.3 Reference Chromatography Parameter

Mobile phase: 5.0 %(V/V) methanol (4.4), 0.20g/100ml Octane sulfonate (4.2), 0.25% Triethylamine solution, adjust pH of the mobile phase to 3.0 with Galacial acetic acid (4.3) and filter it through 0.45μm membrane filter.
Column: C18 150*4.6mm, 5um, or equivalent
Fluorescence detector: excitation wavelength: 293nm, emission wavelength: 395nm.
Inject volume: 10ul
Flowrate: 1.00 ml/min

6.4 Quantitative analysis (external standard method)

Inject a certain volume of standard working solution (4.8.3) to HPLC to get area (or height) Ai of peak of compound i; Inject equal volume of sample solution (6.2.4) to HPLC and get area (or height) Bi of peak of compound i.

7 Calculation and Expression

The content of Vitamin B6 in sample solution, expressed by mass fraction microgram per hundred grams (ug/100g), calculated using formula (1):

Vitamin B6 content of sample,

$X=X_1+X_2\times1.012+X_3\times1.006$…… (1)

In this formula,

X – The content of Vitamin B6 in sample, the unit is microgram per hundred grams ug/100g)

X_1 – The content of pyridoxine in sample, the unit is microgram per hundred grams (ug/100g)

X_2 – The content of pyridoxal in sample, the unit is microgram per hundred grams (ug/100g)

X_3 – The content of pyridoxamine in sample, the unit is microgram per hundred grams (ug/100g)

1.012—— coefficient which the content of pyridoxal converted into pyridoxine

1.006——coefficient which the content of pyridoxamine converted into pyridoxine.

Note: The content of Vitamin B6 in sample solution, expressed by pyridoxine.

The content of compound in sample, expressed by mass fraction microgram per hundred grams (ug/100g), calculated using formula (2):

$$X_i=\frac{c_{si}\times B_i\times V\times100}{m\times A_i\times1000}$$ ……(2)

In this formula,

X_i——The content of compound in sample, the unit is microgram per hundred grams (ug/100g)

B_i—— the peak area (or height) of sample solution gotten from 6.4.

A_i——the peak area (or height) of standard working solution gotten from 6.4.

C_{si}—— the concentration of standard working solution, the unit is microgram per milliliter (μg/mL)；

m——Sample weight, the unit is gram (g)

V——the volume of sample solution, the unit is milliliter (mL).

The result is the arithmetical mean of two independent tests, and reserved one decimal digit.

8 Precision

Absolute difference of two independent test results should not exceed 10% of arithmetical mean under the repeated test condition.

END TRANSLATION